



RECEIVED

APR 25 2001

CASE AG/W-21900/AC 509

CERTIFICATE OF MAILING

TECH CENTER 1600/2900

I hereby certify that this paper (along with any paper referred to as being attached or enclosed) is being deposited with the United States Postal Service on the date shown below with sufficient postage as first class mail in an envelope addressed to the: Assistant Commissioner for Patents, Washington, D.C. 20231.

Anna R. Maddalena      Anna R. Maddalena      4/19/01  
Type or print name      Signature      Date

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF

Group Art Unit: 1619

SIMON A. H. ROSE ET AL

Examiner: R. Bawa

APPLICATION NO: 09/361,816

FILED: JULY 27, 1999

FOR: PROCESS FOR IRRIGATION OF SOIL  
WITH WATER AND COMPOSITIONS  
WHICH PROVIDE FERTILIZATION AND  
SOIL STABILIZATION BENEFITS

Assistant Commissioner for Patents

Washington, D.C. 20231

CLAIM OF PRIORITY UNDER 35 USC 119

Sir:

Applicants in the above-entitled application by their attorney hereby claim priority under the International Convention of British application No. 9816784.4, filed on July 31, 1998. This application is acknowledged in the Declaration of the instant case.

A certified copy of said British application is submitted herewith.

Respectfully submitted,

Ciba Specialty Chemicals Corporation  
Patent Department  
540 White Plains Road  
P.O. Box 2005  
Tarrytown, NY 10591-9005  
(914) 785-7124  
DRC/am/  
Encl. (1)

David R. Crichton  
David R. Crichton  
Attorney for Applicants  
Reg. No. 37,300



The  
Patent  
Office

RECEIVED # 9

APR 25 2001

INVESTOR IN PEOPLE  
TECH CENTER 1600/2900

The Patent Office  
Concept House  
Cardiff Road  
Newport  
South Wales  
NP9 1RH

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears an amendment, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

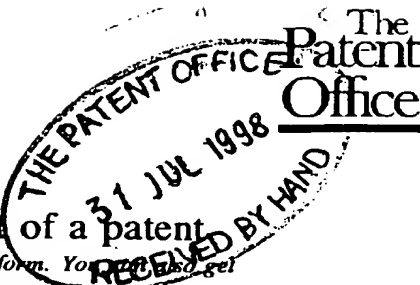
Signed

*AmBrewer*

Dated 20 May 1999.

# Patents Form 1/77

Patents Act 1977  
(Rule 16)



03AUG98 E380285-6 D02890  
P01/7700 25.00 - 9816784.4

## Request for the grant of a patent

(See the notes on the back of this form. You must also get an explanatory leaflet from the Patent Office to help you fill in this form)

The Patent Office

Cardiff Road  
Newport  
Gwent NP9 1RH

31 JUL 1998

1. Your reference

PRL04138GB

2. Patent application number

(The Patent Office will fill in this part)

**9816784.4**

3. Full name, address and postcode of the or of each applicant (underline all surnames)

Allied Colloids Limited

P O Box 38  
Low Moor  
Bradford  
West Yorkshire  
BD12 0JZ  
England

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

England

304 253001

4. Title of the invention

Soil Treatment Compositions  
and Their Use

5. Name of your agent (if you have one)

GILL JENNINGS & EVERY

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Broadgate House  
7 Eldon Street  
London  
EC2M 7LH

PATENT DEPARTMENT  
ALLIED COLLOIDS LTD  
P.O. Box 38  
LOW MOOR  
BRADFORD  
WEST YORKSHIRE  
BD12 0JZ

Patents ADP number (if you know it)

745002

requested by letter 5.10.98

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number  
(if you know it)

Date of filing  
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing  
(day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

YES

- a) any applicant named in part 3 is not an inventor
  - b) there is an inventor who is not named as an applicant, or
  - c) any named applicant is a corporate body.
- See note (d))

# Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form.  
Do not count copies of the same document

## Continuation sheets of this form

Description 19

Claim(s) 2

Abstract 1

Drawing(s)

10. If you are also filing any of the following, state how many against each item.

### Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11. For the Applicant  
Gill Jennings & Every

I/We request the grant of a patent on the basis of this application.

Signature

Date

*Alice Samuels*

31 July 1998

12. Name and daytime telephone number of person to contact in the United Kingdom

SAMUELS, Lucy Alice  
0171 377 1377

## Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

## Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
- Write your answers in capital letters using black ink or you may type them.
- If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- Once you have filled in the form you must remember to sign and date it.
- For details of the fee and ways to pay please contact the Patent Office.

ALLIED COLLOIDS LIMITED

PRL04138GB

SOIL TREATMENT COMPOSITIONS AND THEIR USE

This invention relates to compositions for soil treatment which can provide both fertilisation and soil stabilisation benefits whilst being easy to handle using conventional equipment, and their use in irrigation methods.

It is well known in agriculture to apply various inorganic fertilisers to growing areas. The growing areas may be crop areas, which can be very large, or smaller growing areas such as those in greenhouses. The fertilisers can be supplied in various forms, in particular as solid compositions, or as suspensions or solutions of the fertiliser in a liquid. Fertiliser solutions are generally supplied by the manufacturer as an aqueous concentrate in large batches of size around 1 ton in weight. The solutions contain high concentrations, often 10 to 80 wt.% (dry solids), of dissolved inorganic fertiliser. It is usual to dose the concentrated fertiliser solution into irrigation water so that the solution is diluted and the fertiliser is applied to the soil during irrigation.

Various irrigation systems have been devised for convenient dosing of concentrated fertiliser solution. For instance, large scale irrigation is often carried out by spraying water onto a crop area. Spray pumps are well known which spray water from a spray manifold onto the crop area and which are designed so that concentrated fertiliser solution can be dosed into the pump, mixed with the water and thus diluted before the water is sprayed onto the crop area.

It is also known that some irrigation systems such as furrow irrigation and spray irrigation, as well as naturally occurring processes, can lead to erosion of the soil. It is known to use various polymers as soil stabilisers by applying them to the soil in aqueous solution. This stabilises the soil against erosion.

Polymers of acrylamide and other ethylenically unsaturated monomers have been used as soil stabilisers. It has been generally accepted that the polymers which give optimum soil stabilisation have relatively low anionic content (eg 5 to 30 wt.%) and also have relatively high intrinsic viscosity, for instance above 15 dl/g. Such polymers tend to form viscous aqueous solutions unless used at low concentration. Normal industrial practice is to mix the polymer in powder or reverse phase emulsion form with water in special dissolution apparatus so as to form an aqueous solution of the polymer having a concentration of, for instance, 0.2 to 2 wt.%. This solution can then be dosed into the irrigation water.

US 3,798,838 describes a method of reducing the water permeability of soils by contacting them with an aqueous solution containing a water-soluble nutrient and a water-soluble partially hydrolysed polyacrylamide. The solution which is contacted with the soil may not contain more than 2 wt.% polymer or 5 wt.% nutrient. Preferred amounts are much lower, for instance not more than 0.3 wt.% polymer and not more than 0.05 wt.% nutrient. In the examples a dilute solution of this type is produced. The concentration of polyacrylamide is 70 ppm. The level of nutrient is 1.15 wt.%. The publication states that the polymer and the nutrients should be dissolved in the irrigation water. Thus the polymer and nutrient would be added separately directly to the irrigation water to give the dilute solution applied to the soil. There is no suggestion to provide a more concentrated solution containing both components. If polymer were to be included in a solution with the concentrated aqueous solution of fertiliser, the amount of polymer required to give an appropriate concentration on the soil (after dilution to the degree required for the fertiliser) would be high, ie at least 2 wt.%. The polyacrylamide used in the Examples has 20 to 30% of its amide groups hydrolysed, ie 20 to 30% anionic content. We have found that polymers of this type produce

a highly viscous solution at such concentrations. As a result the solution cannot be processed in the same apparatus as standard fertiliser solution. Therefore the system of US 3,798,838 is limited to separate addition of the polymer and the fertiliser to the irrigation water. This necessitates, in commercial practice, the provision of two separate addition points and make-up systems.

Other publications describe various compositions which contain both a conventional low anionic content soil conditioner and a fertiliser, such as EP-A-586,911 and JP-A-74/144087. JP-A-07/322753 and JP-A-07/099833 each describe soil conditioning compositions containing acrylamide polymers. Aqueous compositions containing polymer and fertiliser are not disclosed. JP-A-73/037738 also discloses a composition suitable for spraying comprising fertiliser, polyacrylamide and seeds, together with a collection of other materials such as cellulose and lignin sulphonic acid. JP-A-51/124578 also discloses a liquid fertiliser composition containing phosphorus fertiliser and a copolymer of acrylamide and unspecified amounts of potassium acrylate.

It would be desirable to be able to provide a combined composition which gives both fertilisation and soil stabilisation benefits, so as to avoid the need for separate addition points, but to be able to use dosing equipment currently in place for irrigation systems, in particular equipment in place for processing fertiliser solution. Such systems include pivots, wheel-lines and drip systems.

According to the invention we provide an aqueous soil treatment composition comprising water and, in solution,

(a) an ionic (eg inorganic or urea-containing) water-soluble fertiliser at a concentration of at least 10 wt.%, and

(b) a water-soluble anionic polymer which has intrinsic viscosity at least 6 dl/g and is formed from

water-soluble monomer or monomer blend of which at least 40 wt.% is anionic monomer.

Thus we provide a single pack product which gives both fertilisation and soil stabilisation benefits. The product is thus a growth-promoting composition. It can be produced by adding polymer to a standard concentrated solution of fertiliser, at a concentration sufficiently high to provide appropriate polymer concentration on the soil after dilution of the concentrated solution in the standard manner for fertiliser solutions. We find that the defined anionic polymers having relatively high anionic monomer content give rise to fewer of the viscosity problems which we observe with the standard, non-ionic or low anionic content, soil stabilisation polymers. In fact, in the solution of fertiliser they have relatively low viscosities which can even be below 1,000 cps, in particular below 500 cps. Thus the viscosity of the fertiliser solution is not increased to an inconvenient degree and the aqueous composition of the invention can be processed using the equipment which is in place for processing of solution of fertiliser alone. A further advantage of the defined polymers is that their low viscosity in the composition means that they can be added without difficulty to the fertiliser solution at the fertiliser production plant. The prevention of excessive viscosity allows the compositions to be processed at the fertiliser production plant using conventional equipment.

We believe that the interaction between the fertiliser, which is an ionic salt, and the defined anionic polymer is important in the invention. We believe that the polymer dissolves in the aqueous composition but in the presence of the fertiliser salt the polymer molecules are not fully extended, thus reducing the viscosity of the composition. However, as the composition is diluted during an irrigation process by mixing with the irrigation water, the concentration of fertiliser is reduced and the polymer molecules are able to extend fully and act as soil



stabilisers. Similarly, if the composition is placed directly onto an area of soil, the fertiliser gradually dissolves into the soil, thus reducing its concentration around the polymer molecules, which can again extend and act as soil stabilisers.

The invention provides an aqueous composition in which the defined polymer (b) is dissolved. That is, the polymer is taken into the solution such that substantially no visible solid material remains. For some combinations of polymer and fertiliser, some solid lumps or other material visible to the naked eye may remain. These may be filtered out before use. It is not necessary that the polymer molecules be fully extended and solvated within the solution. In fact, we believe that it is useful in the invention that the polymer molecules, although dissolved, are not fully extended in the solution. They thus give rise to lower viscosity than they would if fully extended.

The polymer must be water-soluble and in particular is soluble in an aqueous solution of fertiliser having the same concentration of fertiliser as the final aqueous composition which is desired to be produced. Generally the polymer is substantially linear and is not cross-linked.

The polymer has intrinsic viscosity at least 6 dl/g. In this specification intrinsic viscosity is measured by suspended level viscometer at 20°C in 1 M sodium chloride buffered to pH 7. That is, it is of sufficiently high molecular weight to give a soil stabilisation effect and is not a low molecular weight material which would act as a dispersant.

Preferably IV is at least 8 dl/g, more preferably at least 9 dl/g. It may be up to for instance 30 dl/g but generally we find that the optimum combination of low viscosity of the composition and soil stabilisation performance is given by polymers having IV not more than about 20 or 18 dl/g. Preferably IV is not more than 16, more preferably not more than 15 dl/g. Particularly

preferred IV ranges are from 9 to 13 dl/g, especially 10 to 12 dl/g.

The polymer is formed from water-soluble monomer or monomer blend, usually water-soluble ethylenically unsaturated monomer. The anionic content, ie the proportion of anionic monomer in the monomer blend used to form the polymer, is at least 40 wt.%, preferably at least 45 or at least 50 wt.%. In particular it is at least 55 wt.%. It can be up to 100 wt.%, but is preferably not more than 90 wt.%, more preferably not more than 80 wt.%. Particularly preferred polymers have anionic content in the range 65 to 85 wt.%.

The monomer blend used to form the polymer comprises at least 40 wt.% anionic monomer. This may be any suitable anionic ethylenically unsaturated monomer. It can be a sulphonic monomer, often as sodium or other alkali metal salt, for instance acrylamido propane sulphonic acid (AMPS). It is generally preferred that the anionic monomer is an ethylenically unsaturated carboxylic monomer, in particular acrylic or methacrylic monomer. Salts of acrylic acid are preferred, for instance ammonium or alkali metal, in particular sodium, salts.

The polymer may contain small amounts of cationic monomer, for instance up to 20 wt.% or 10 wt.% but usually the content of cationic monomer is substantially zero.

Generally the anionic monomer is copolymerised with non-ionic monomer, usually ethylenically unsaturated water-soluble non-ionic monomer such as acrylamide or methacrylamide, preferably acrylamide.

Particularly preferred polymers are copolymers of acrylamide with sodium acrylate.

The polymer is included in the composition in an amount such that when the composition is diluted for irrigation, to the degree of dilution conventionally used for fertiliser solutions, it provides adequate polymer concentration on the soil to give acceptable soil stabilisation performance. Polymer concentration in the

aqueous soil treatment composition of the invention is generally from 0.2 to 10 wt.%, in particular at least 0.5 or at least 1 wt.%. Preferably it is at least 1.5 wt.%. Often it is not more than 7 wt.%, and particularly preferred compositions contain from 2 to 5 wt.% polymer.

The composition also contains an ionic fertiliser in solution. Thus the fertiliser must be water-soluble. Fertilisers for use in the invention can also be described as inorganic or urea-containing fertilisers. The fertiliser is present in a concentration at least 10 wt.% and is usually as high as is convenient, for instance at least 20 wt.%. Preferably it is at least 30 wt.%. It can be as high as 70 or 80 wt.% but is usually not more than 60 wt.%. Preferred concentration ranges are 30 or 40 to 55 wt.%, for instance around 50 wt.%.

Urea and any of the known inorganic fertiliser materials can be used, which provide nitrogen, phosphorus and/or potassium, either alone or in a mixture. These contain ionic salts and include ammonium nitrate, ammonium sulphate, mono ammonium phosphate, diammonium phosphate, mono potassium phosphate, dipotassium phosphate, polyphosphate salts, potassium chloride, potassium sulphate and calcium nitrate.

Examples include urea/ammonium nitrate (32-0-0), potassium chloride (0-0-10), ammonium sulphate (8-0-0-9S), a blend of calcium and ammonium nitrate, and blended fertilisers with the following analyses: 18-0-8, 10-0-10, 3-18-18, 0-0-25-17S, 10-10-10 and 14-2-10-2+2.5% organic matter (OM).

The invention is particularly useful when it is known to be desired to use a particular fertiliser on the relevant soil area. The invention allows the operator to select a polymer which gives, in combination with that chosen fertiliser, an appropriate balance of viscosity of the final composition and soil stabilisation performance. We find that any given polymer will not necessarily give optimum results for all fertilisers, but generally we find

that polymers of the types defined in the invention are capable of giving an acceptable range of results in combination with a range of fertilisers. The particular intrinsic viscosity and anionic content within the claimed  
5 ranges which are optimum for combination with any given fertiliser, as well as the concentration of polymer and the concentration of fertiliser, can be established by experimentation.

In particular we find that for use with UAN (32-0-0) solution polymers of anionic content 60 to 70 wt.% and IV  
10 9 to 11 dl/g, for instance anionic content 65 wt.% and IV 10 dl/g, give low viscosity and good soil stabilisation performance. Polymers of higher anionic content, for instance from 90 to 100 wt.%, and slightly higher intrinsic  
15 viscosity, for instance 12 to 14 dl/g, give higher viscosity with the same fertiliser solution although the compositions can still be handled, and show particularly good stabilisation performance.

Polymers of anionic content 60 to 70 wt.% and IV 9 to  
20 11 dl/g give low viscosity and good soil conditioning performance in combination with fertilisers 8-0-0-9S (ammonium sulphate) and 0-0-10 (KCl).

In combination with 8-0-0-9S, polymers of anionic content 90 to 100 wt.% and IV 12 to 14 dl/g give somewhat  
25 higher viscosity, but again the compositions remain handleable, and show excellent stabilisation performance. These polymers similarly give compositions of high but acceptable viscosity and excellent stabilisation with fertilisers 10-0-10 and 10-10-10.

30 With fertilisers 18-0-18, 10-0-10, 10-10-10 and 14-2-10-2+2.5% OM, polymers of anionic content 60 to 70 wt.% and IV 9 to 11 dl/g also give reasonable viscosity and stabilisation performance.

35 Polymers of anionic content 45 to 50 wt.% (eg about 47 wt.%) and IV 10 to 12 dl/g (eg about 11 dl/g) also give reasonable viscosity and stabilisation performance with 0-0-10 (KCl).

The aqueous composition of the invention can be made in any convenient manner. For instance polymer may be added to water followed by fertiliser or the two may be added simultaneously. Alternatively, fertiliser may be added to a preformed solution of polymer. However, generally it is preferred that the polymer is added to a preformed solution of the fertiliser. In particular it is preferred that the polymer is added in solid form, ie powder or bead. It is possible to add it in other forms, such as reverse phase dispersion, but solid is preferred. Preferred solids are in the size range 70 to 2,000  $\mu\text{m}$  and are made in standard manner, for instance by gel or bead polymerisation followed by comminution and if necessary drying.

The polymer, if added as powder, can be added using known systems such as those described in US 4,086,663 and WO94/19095 and systems available from Allied Colloids under the name JetWet (trade mark).

Thus in the invention we provide a process for the production of an aqueous composition of the invention comprising providing a preformed aqueous solution of the fertiliser (a) and mixing into it polymer (b) in powder form.

The thus formed composition should have viscosity which renders the composition easy to handle. It should in particular be easy to handle (ie preferably pumpable, pourable or sprayable) in the equipment which is presently used for addition of concentrated fertiliser solution to irrigation water and for application of concentrated fertiliser solution alone to soil. The polymer may be added to the fertiliser at the facility of the fertiliser manufacturer and thus the viscosity should be low enough that the resulting solution can be handled by equipment presently in place in such facilities. Preferably viscosity is below 4,000 cPs, more preferably not more than 3,000 cPs. In particular it is not more than 2,000 and especially not more than 1,000 cPs. Particularly

preferably it is not more than 600 cPs. Usually it is at least 100 cPs, and often at least 200 cPs. Particularly preferred compositions have final viscosity of from 200 to 500 cPs. In this specification viscosity is measured using  
5 a Brookfield LVT viscometer at 30 rpm using spindle 4.

Thus aqueous composition of the invention may be applied directly to soil as a fertiliser/soil conditioner composition. However, the composition of the invention is intended particularly as a concentrate for use in  
10 irrigation, in which processes it will be diluted and applied to soil areas. Thus it may contain any materials known for inclusion in such compositions. It generally does not contain additional materials such as oxidising agents, reducing agents, soil materials or seed materials.

15 The invention also provides a soil treatment process comprising irrigating an area of soil with water to which has been added an aqueous soil treatment composition of the invention.

Suitable irrigation processes include drip irrigation, furrow irrigation and spray irrigation. In particular the  
20 composition is suitable for use in processes of spray irrigation where irrigation water is pumped to a spray manifold and sprayed over a very large crop area, for instance at least one hectare (ha), and even up to 100 ha. Such methods comprise pumping water through feed ducting  
25 and a mixing zone to a spray manifold supplying one or more spraying devices by which the water is sprayed onto the crop area to be irrigated, and the aqueous composition of the invention is metered into the water at or before the  
30 mixing zone. Processes of this general type are described in our unpublished PCT Application No. PCT/GB98/01763. In that application it is essential to supply polymer in the form of a dispersion of water-soluble polymer particles in a liquid. In the present case the polymer is of course  
35 included in the solution of fertiliser which is metered in the standard manner and thus this system is not necessary in the present case. However, other features of the

system described in that application can however be applied herein.

The invention will now be illustrated with reference to the following examples.

5    **Examples**

In the following examples various fertilisers were used, as follows:

Urea/Ammonium Nitrate (32-0-0)

Muriate of Potash (0-0-10)

10    Sulfate of Ammonia (8-0-0-9S)

Calcium Ammonium Nitrate

Blended fertilisers with analysis:

18-0-8

10-0-10

15    3-18-18

0-0-25-17S

10-10-10

14-2-10-2+2.5% OM

**Example 1**

20    Several polymers, of medium to high anionic charge and in dry powder form, were evaluated initially for their solubility and viscosity in concentrated solutions of fertiliser. This was assessed by adding the polymer powder to the fertiliser solution and tumbling for 4 to 5 hours at  
25    slow medium speed. The polymer powder was added in such an amount as to give a concentration in the composition of about 2 wt.%.

The viscosities of the resultant products were measured using a Brookfield LVT Viscometer, spindle 4 at 30  
30    rpm.

If the resultant product was not highly viscous, ie was pourable, the flocculation and stabilisation tests were performed. Both of these tests give an indication of the soil stabilisation efficacy of the relevant polymer in  
35    combination with the relevant fertiliser. These tests were evaluated using 34 ppm hardness water, to simulate the

irrigation water commonly used in the USA (a region in which large-scale irrigation is widely practised).

The polymers used were as follows:

- 5 Polymer A: Copolymer of 14.6 wt.% sodium acrylate and 85.4 wt.% acrylamide, IV 16 dl/g.
- Polymer B: Copolymer of 65 wt.% sodium acrylate and 35 wt.% acrylamide, IV 10 dl/g.
- Polymer C: Copolymer of 95.5 wt.% sodium acrylate and 4.5 wt.% acrylamide, IV 13 dl/g.
- 10 Polymer D: Copolymer of 40 wt.% sodium AMPS and 60 wt.% acrylamide, IV 18 dl/g.
- Polymer E: Copolymer of 20 wt.% sodium AMPS and 80 wt.% sodium acrylate, of low IV.

15 The fertiliser solutions were all used as supplied by the manufacturer, with varying solids content of from 10 to 80 wt.% solids.

#### Flocculation

20 The 34 ppm hardness water was added to a 1,000 ml stoppered measuring cylinder (~950 g). The solution of polymer and fertiliser was then added at 15 ppm before the remainder of the water was added, to total 1,000 g. The cylinder was inverted several times to allow the product to disperse. Soil fines (< 500  $\mu$ m size particles of a clay loam soil), 20 g, were added to the cylinder and the cylinder shaken vigorously for 20 seconds, before leaving to stand for a further 2 minutes, to allow flocculation to occur. An aliquot was then extracted from a predetermined depth and the turbidity measured, to assess the effectiveness of the product. The clearer the water, the more effective the product.

30

#### Stabilisation

35 The test above was repeated, using standardised (sieved and dried) soil crumbs and not fines. In this test, the cylinder was not shaken vigorously, but was inverted 10 times in 20 seconds, before leaving to stand for 2 minutes. The turbidity was then measured.



A control was also performed for the flocculation and stabilisation tests, where the water alone was used, without any polymer present.

5 The results in Table 1 below show the effect of the polymers on flocculation/soil stabilisation. The stabilisation test was only performed on products which were pourable and which produced good flocculation results.

**Table 1**

Fertiliser	2% Polymer	Viscosity (cPs)	Turbidity NTU (Flocculation)	Turbidity NTU (Stabilisation)
Control	---	---	758.7	135
UAN 32	A	11,030	171.3	---
UAN 32	B	1,510	216.3	38.3
UAN 32	C	4,140	177.3	52
UAN 32	D	6,780	528	---
8-0-0-9S	B	640	285.3	---
8-0-0-9S	C	3,790	76.7	18
8-0-0-9S	E	90	798	---

It can be seen from this example that Polymer B, which has anionic content of 65 wt.% and IV 10 dl/g, is particularly suitable for providing low viscosity, and thus pumpable and sprayable, compositions. In particular it gave adequately low viscosity with two different fertilisers, UAN 32 and 8-0-0-9S. In this particular test, flocculation in the 8-0-0-9S solution was not optimum.

It can also be seen that Polymer C, which has a higher anionic content of 95.5 wt.%, gives higher viscosities than Polymer B, but lower than those of Polymers A and D in UAN 32 solution. In addition, it gives good flocculation and stabilisation performance.

Polymers A and D gives rather high viscosity values and Polymer E does not give good flocculation.

#### Example 2

In this Experiment the effects of the IV and anionic content of various polymers in various fertiliser solutions are assessed.

#### FED Experiment

A factorial experiment design, using solid grade acrylamide/sodium acrylate polymers (added at 2% w/w to the concentrated fertiliser solution), covering a range of IV's from 10 to 24 and anionic contents of 0 to 100%, was evaluated. In each case, the viscosity and flocculation of the resultant solutions were measured as in Example 1 above. This was performed with a potassium chloride solution (10% K<sup>+</sup>), which was similar to the 0-0-10(KCl) fertiliser solution. A control was performed, using 15 ppm of the KCl solution, without added polymer.

The factorial experimental design results are in Table 2 below. Some products are listed twice. This is because two different batches were assessed. It will be seen that polymers which can give rather high viscosities can also give variable results from batch to batch.

Table 2

Polymer	Anionic (%)	IV	Viscosity (cPs)	Flocculation (NTU)
Control	---	---	---	645
F	28	12	5,050	327
G	14.25	18	12,500	126
B	65	10	350	240
B	65	10	375	148
H	47	11	1,250	281
H	47	11	3,575	248
C	95.5	13	4,900	120
I	75	20	7,000	180
A	14.6	16	8,625	232
J	48	24	11,375	407
Q	0	16	8,750	39
K	14.9	24	V.Viscous	---
L	42	16	5,975	284
L	42	16	6,150	324
H	47	16	5,600	216
H	47	16	9,000	267
N	84	19	6,500	150
P	100	13	6,750	426

5

10

15

20

It can be seen from these results that Polymer B again gives a low viscosity and good flocculation performance this fertiliser solution.

5 Polymers F, G, A, Q and K all have anionic content below 40 wt.% and give rather high viscosity values, in particular Polymers G, A, Q and K. From the point of view of achieving low viscosity in combination with adequate flocculation performance, it appears that a value of IV of from about 9 or 10 to about 13 or 14 tends to be optimum in  
10 combination with anionic content of at least 40 wt.%. It also appears that anionic content of from about 55 to 80 wt.% may be optimum.

These tests were carried out with a fertiliser solution similar to 0-0-10 (KCl) solution. Certain of the  
15 polymers may give better performance with other fertiliser types.

### Example 3

In this Example flocculation tests were also performed on the optimum polymers determined in Example 1 for each  
20 fertiliser, using a different soil type. These were assessed using soil of a loamy sand nature.

The optimum products to use in UAN (32-0-0) and 8-0-0-9S fertilisers were found in Example 1 to be Polymer B and Polymer C respectively. These were evaluated with a second  
25 soil type, a loamy sand. The 8-0-0-9S was also assessed with Polymer B, as this produced a much less viscous solution (640 cPs), than Polymer C (3790 cPs). Results were as follows.

**Table 3**

Fertiliser & Polymer	Flocculation (NTU)	Control (NTU)
KCl + B	201 (194)*	519
UAN + B	267 (216)*	574
8-0-0-9S + C	78 (77)*	507
8-0-0-9S + C	335 (285)*	507

\*(The figures in brackets are the original turbidity results for flocculation, when performed on the first soil type in Example 1, a clay loam).

It can be seen that although flocculation was in general not as efficient as on the previously used soil, it was nevertheless effective.

**Example 4**

The solubilities of Polymer B and Polymer C (the optimum polymers determined in Example 1) were assessed at 2% w/w in various fertiliser solutions. The viscosities of the polymers in fertiliser solutions were measured, if the polymers were soluble, and so was the flocculation efficiency. Results are below in Table 4.

**Table 4**

Fertiliser	2% Polymer	Viscosity (cPs)	Flocculation
18-0-8	B	1,000	155
18-0-8	C	3,600*	---
10-0-10	B	900	151
10-0-10	C	3,950	124
10-10-10	B	950	306
10-10-10	C	3,850	297
0-0-25-17S	B	*	
0-0-25-17S	C	10,000	---
14-2-10-2+2.5% OM	B [Mag 525]	1,500	205
14-2-10-2+2.5% OM	C [Alclar W5]	7,500	---

\* - large quantity of undissolved polymer after tumbling overnight.

Both products were insoluble in the 3-18-18 fertiliser solution.

These results again demonstrate the general usefulness of Polymer B, which gave low viscosity solutions and good flocculation for a range of fertilisers. Polymer C could also give reasonable viscosity with certain fertilisers such as 18-0-8, 10-0-10 and 10-10-10.

CLAIMS

1. An aqueous soil treatment composition comprising water and, in solution,
  - (a) an ionic water-soluble fertiliser in an amount of  
5 at least 10 wt.%, and
  - (b) a water-soluble anionic polymer which has intrinsic viscosity at least 6 dl/g and is formed from water-soluble monomer or monomer blend of which at least 40 wt.% is anionic monomer.
- 10 2. A composition according to claim 1 in which the polymer (b) has intrinsic viscosity from 8 to 18 dl/g.
3. A composition according to claim 1 or claim 2 in which the polymer (b) has intrinsic viscosity of from 9 to 12 dl/g.
- 15 4. A composition according to any preceding claim in which the polymer (b) is formed from water-soluble monomer or monomer blend comprising at least 50 wt.% anionic monomer.
5. A composition according to any preceding claim in  
20 which the polymer (b) is formed from water-soluble monomer blend comprising from 60 to 80 wt.% anionic monomer and from 40 to 20 wt.% non-ionic monomer.
6. A composition according to any preceding claim in which the polymer (b) is a copolymer of acrylamide with an  
25 alkali metal salt of acrylic acid.
7. A composition according to any preceding claim in which the polymer (b) is present in an amount of from 2 to 5 wt.%.
8. A composition according to any preceding claim in  
30 which the fertiliser (a) is present in an amount of from 20 to 60 wt.%.
9. A composition according to any preceding claim which has a viscosity of not more than 4,000 cps, preferably not more than 1,000 cps.
- 35 10. A composition according to any preceding claim which has a viscosity of from 200 to 500 cps.



11. A composition according to any preceding claim in which the polymer (b) has been added to the composition in the form of a powder.

5 12. A soil treatment process comprising irrigating an area of soil with water to which has been added an aqueous soil treatment composition as defined in claim 1.

13. A process according to claim 12 in which the irrigation is by furrow irrigation, drip irrigation or spray irrigation.

10 14. A process according to claim 12 in which water is pumped through feed ducting and a mixing zone to a spray manifold supplying one or more spraying devices by which the water is sprayed onto a crop area and the aqueous soil treatment composition is metered into the water at or  
15 before the mixing zone.

15. A method for the production of an aqueous soil treatment composition as defined in claim 1 comprising providing an aqueous solution of fertiliser (a) and mixing with it polymer (b) in powder form.

20 16. A process according to claim 12 or a method according to claim 15 in which the aqueous soil treatment composition has any of the additional features of claims 2 to 10.

**ABSTRACT****SOIL TREATMENT COMPOSITIONS AND THEIR USE**

5       The invention provides aqueous soil treatment  
compositions comprising water and dissolved ionic water-  
soluble fertiliser in an amount of at least 10 wt.% and  
dissolved water-soluble anionic polymer having IV at least  
6 dl/g and ionic content at least 40 wt.%. Such  
concentrates can have low viscosity and be pourable and be  
10       used as concentrates for dilution in irrigation processes.